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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
SHINJI SAKASHITA : EXAMINER: VANESSA VELASQUEZ
SERIAL NO: 10/522,779 :
FILED: FEBRUARY 1, 2005 : GROUP ART UNIT: 1793
FOR: TITANIUM ALLOY MATERIAL :
HAVING SUPERIOR HYDROGEN
ABSORPTION RESISTANCE

DECLARATION UNDER 37 C.F.R. §1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Shinji SAKASHITA who deposes and states that:

1. I have a doctorate degree in engineering, which was conferred upon me in 2002 by Tohoku university located in Sendai, Japan.
2. I have been employed by Kobe Steel, Ltd. Since 1993, and I have a total of 15 years of work and research experience in the field of materials engineering.
3. I understand the English language or, at least, the contents of this Declaration were made clear to me prior to executing the same.
4. The following experiments were carried out by me or under my direct supervision and control.

Titanium alloys shown in Table 1 were manufactured in a vacuum ark melting furnace using pure metals such as pure Fe, Al, and Ti of JIS class 1 (corresponding to ASTM Gr. 1) as starting material to produce the ingots (about 500 g) of titanium alloy shown in

Table 1. After thermal refining annealing (1000°C, 2 hours), the ingot was formed into a sheet having a thickness of 4.2 mm by hot rolling (at 800 to 900°C). After removing scale by pickling, the sheet was cold rolled to produce a sheet having a thickness of 1.0 mm (thickness reduction, 75%), and this sheet was annealed in vacuum (800°C, 1 hour). After the vacuum annealing, the sheet was anodized in 1 vol% aqueous solution of phosphoric acid at a temperature of 30°C by applying a voltage of 20 V, and then oxidized in atmosphere at 400°C for 10 minutes (test piece A). For this test piece, the Al concentration distribution in the thickness direction from the surface of the test piece was measured by Auger electron spectroscopy, and thickness of the oxidized film and the average Al concentration and thickness of the Al concentration layer were determined. The results are shown in Table 2. The proportion of the crystalline part (crystallinity) and crystal structure of the crystalline part were evaluated by electron diffraction. The results are also shown in Table 2.

In order to evaluate the hydrogen absorption resistance of the titanium alloy in contact with the steel material, a 30 mm x 30 mm test piece was cut out from the two surface treated titanium sheets as described above, and a hole having a diameter of 5 mm was formed in the center of the test piece. The test piece was stuck to a carbon steel (JIS SPCC) of the identical shape, and after fastening the laminate by titanium bolts and nuts, the laminate was immersed in a corrosive solution to evaluate its hydrogen absorption properties. The corrosive solution used was 3 wt% NaCl aqueous solution (boiling), and the immersion time was 2 months. The amount of hydrogen absorption after the test was measured by the melting method.

5. The following Results were obtained:

The results of the hydrogen absorption resistance evaluated for the titanium alloy in contact with the steel material are shown in Table 2. In the case of the test piece A which had been anodized and oxidized in atmosphere, the difference of the Al concentration between the

Al concentration layer and the substrate was 0.25%, and the hydrogen absorption properties were far from sufficient, compared to the present invention (Nos. 48, 49, 55 – 59 in Table 3 of the specification).

Table 1

Al	Fe	Mo	Ni	Nb	Mn	Ti
0.56	0.07	0.06	0.14	0.06	0.02	bal.

Table 2

No.	Surface oxide layer			Al concentration layer		Absorbed hydrogen (*2)
	Thickness (nm)	Crystallinity (%)	Structure (*1)	Al (mass%)	Thickness (um)	
A	1.8	50.2	R	0.81	0.08	E

*1 Crystal structure R: rutile structure

*2 Hydrogen absorption: A: less than 10 ppm; B: 10 to 49 ppm; C: 50 to 99 ppm; D: 100 to 499 ppm; E: 500 to 999 ppm; F: 1000 ppm or higher

6. The Al concentration layer of the test piece A had the difference in the Al concentration with the substrate of 0.25%, which was outside the scope of the present invention (at least 0.3%), and the resistance to hydrogen diffusion was insufficient. Presumably, this is the reason for the insufficient improvement of the hydrogen absorption properties.

From the results as described above, it is concluded that formation of the Al concentration layer having the difference of the Al concentration with the substrate of at least 0.3% should provide a titanium alloy having the resistance to hydrogen absorption superior to that of conventional products.

7. I declare further that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United

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States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

8. Further Declarant saith not

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Name: Shinji SAKASHITA
Kobe Steel, Ltd.

August 19, 2008

Date